

What we claim is:

1. A process for preparing a high purity and high concentration hydroxylamine product, the process comprises
 - 5 a. feeding an aqueous feed solution containing a hydroxylammonium salt at a maximum concentration and a stabilizer into a reactor at a low temperature;
 - b. neutralizing the aqueous feed solution with a base to form a slurry until the pH of the resulting slurry reaches in a range of from about 9.0 to about 12.0;
 - c. adding an additional amount of solid of the hydroxylammonium salt to
 - 10 increase the total amount of the hydroxylammonium salt in the reactor before or during neutralization of step b;
 - d. separating the resulting slurry into an aqueous solution phase and a solid phase at a temperature between about -20°C to about 20°C;
 - e. treating the aqueous solution phase in at least one ion exchange column
 - 15 containing at least one ion exchange resin to produce a high purity hydroxylamine solution thereby the resistivity is not lower than about 10 Megohms-cm; and
 - f. concentrating the high purity hydroxylamine solution at a sub-atmospheric pressure to form the high purity and high concentration hydroxylamine product.
2. The process of claim 1, wherein the hydroxylammonium salt is a salt of a mineral acid selected from the group consisting of hydroxylammonium sulfate, hydroxylammonium nitrate, hydroxylammonium chloride and mixtures thereof.
3. The process of claim 2, wherein the hydroxylammonium salt consists essentially
 - 25 of hydroxylammonium sulfate.
4. The process of claim 1, wherein the base is selected from a strong base or a weak base, wherein the strong base is selected from sodium hydroxide, potassium hydroxide and mixtures thereof; and the weak base is selected from ammonia, ammonium hydroxide, volatile alkylamines, cyclic amines and mixtures thereof.
5. The process of claim 4, wherein the base is ammonia.
6. The process of claim 1, wherein the stabilizer is a compound selected from the group consisting of hydroxyanthraquinone, substituted hydroxyanthraquinones, thiourea, substituted thioureas, hydroxyurea, substituted hydroxyureas, aminoquinoline, substituted aminoquinolines, phenanthroline, substituted phenanthrolines, one or more
 - 30 polyhydroxyphenols, cyclohexanediaminetetraacetic acid, thiamine or its derivatives, ethylenediaminetetraacetic acid or its derivatives, and mixtures thereof.

7. The process of claim 6, wherein the stabilizer consists essentially of cyclohexanediaminetetraacetic acid.
8. The process of claim 1, wherein the neutralization is conducted at a temperature in the range of from about 5°C to about 65°C.
- 5 9. The process of claim 1, wherein the neutralization is conducted at a temperature in the range of from about 10°C to about 55°C.
- 10 10. The process of claim 1, wherein the neutralization is conducted at a temperature in the range of from about 15°C to about 45°C.
11. The process of claim 1, wherein the hydroxylammonium salt consists essentially of hydroxylammonium sulfate and the ratio of the additional amount of the solid added to the amount of hydroxylammonium sulfate in the aqueous feeding solution is in the range of from about 0 to about 4.8 on a weight to weight basis.
12. The process of claim 11, wherein the ratio is in the range of from about 0.5 to about 4.6.
- 15 13. The process of claim 11, wherein the ratio is in the range of from about 0.6 to about 4.5.
14. The process of claim 1, wherein separation in step d is conducted by centrifugation, filtration, or combination thereof.
- 20 15. The process of claim 14, wherein the temperature of step d is between about -10°C to about 15°C,
16. The process of claim 14, wherein the temperature of step d is between about 5°C to about 15°C.
- 25 17. The process of claim 1, wherein the base is ammonia, the hydroxylammonium salt consists essentially of hydroxylammonium sulfate, and the aqueous solution phase contains greater than 50% of hydroxylamine after removal of ammonium sulfate and residual ammonia.
18. The process of claim 1, wherein the ion-exchange resin is selected from the group consisting of cation-exchange resin, anion-exchange resin, and mixtures thereof thereby ammonium cation and anions of mineral acids are removed.
- 30 19. The process of claim 18, wherein the anion exchange resin is selected from one or more strong base gel resins, one or more macroporous resins, or mixtures thereof.
20. The process of claim 18, wherein the cation exchange resin is selected from one or more strong acid gel resins, one or more macroporous resins, or mixtures thereof.

21. The process of claim 18, wherein the ion exchange process is carried out in four stages:

- A. feeding the aqueous solution phase into a first column containing the anion exchange resin until breakthrough of salt anions in the effluent;
- 5 B. regenerating the anion exchange resin with sulfuric acid to form a sulfate form followed by converting the sulfate form into an hydroxide form;
- C. feeding the effluent from stage A into a second column containing the cation exchange resin until breakthrough of ammonium ion; and
- 10 D. regenerating the cation exchange resin with sulfuric acid.

22. The process of claim 1, wherein the process is carried out in a continuous moving bed or simulated moving bed ion exchange apparatus.

23. The process of claim 1, wherein the concentration step f is conducted under the sub-atmospheric pressure in the range of from about 6 torrs to about 100 torrs [about
15 0.8 kPa to about 13.3kPa].

24. The process of claim 1, wherein the sub-atmospheric pressure in the range of from about 10 torrs to about 80 torrs [about 1.33 kPa to about 10.7 kPa].

25. The process of claim 1, wherein the sub-atmospheric pressure in the range of from about 10 torrs to about 60 torrs [about 1.33kPa to about 8.0 kPa].

20 26. The process of claim 1, wherein the temperature for the concentration step f is in the range of from about 25°C to about 60°C.

27. The process of claim 1, wherein the temperature for the concentration step f is in the range of from about 30°C to about 50°C.

28. The process of claim 1, wherein the temperature for the concentration step f is in
25 the range of from about 35°C to about 45°C.

29. The process of claim 1, wherein temperature change during neutralization step b is maintained within a range of from about 0 to about 25°C.

30. A process for preparing a high purity and high concentration hydroxylamine product, the process comprises:

- 30 a. feeding an aqueous feed solution containing a hydroxylammonium salt consisting essentially of hydroxylammonium sulfate at a maximum concentration and a stabilizer consisting essentially of cyclohexanediaminetetraacetic acid into a reactor at a low temperature;
- b. neutralizing the aqueous feed solution at a temperature in the range of from
35 about 15°C to 45°C with a base to form a slurry until the pH of the resulting

- slurry reaches in a range of from about 9.0 to about 12.0;
- 5 c. adding an additional amount of solid of hydroxylammonium sulfate wherein the ratio of the additional amount of solid added to the amount of hydroxylammonium sulfate in the aqueous feeding solution is in the range of from 0.6 to 4.5 on a weight to weight basis to increase the total amount of hydroxylammonium sulfate in the reactor before or during neutralization of step b;
- 10 d. separating the resulting slurry by centrifugation and filtration into an aqueous solution phase and a solid phase at a temperature between about 5°C to about 15°C;
- 15 e. treating the aqueous solution phase in a first column containing anion exchange resin to form an effluent followed by treating the effluent from the first column in a second column containing a cation exchange resin to produce a high purity hydroxylamine solution thereby the resistivity is in the range of from about 10 Megohms-cm to about 50 Megohms-cm; and
- f. concentrating the high purity hydroxylamine solution at a sub-atmospheric pressure in the range of from about 10 torrs to about 60 torrs [about 1.33 kPa to about 8.0 kPa] and a temperature in the range of from about 35°C to about 45°C to form the high purity and high concentration hydroxylamine product.
- 20 31. The process of claim 30, wherein the aqueous solution phase contains greater than 50% of hydroxylamine after removal of ammonium sulfate and residual ammonia.